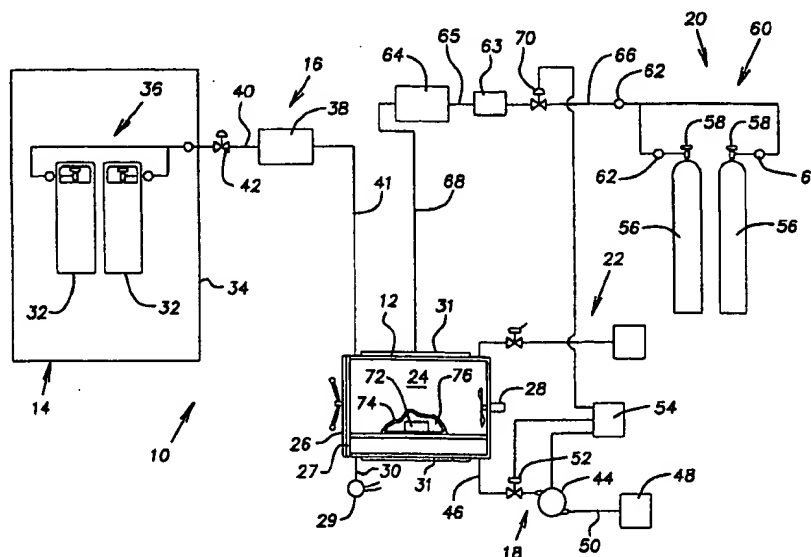




## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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<b>(21) International Application Number:</b> PCT/US99/26367  <b>(22) International Filing Date:</b> 9 November 1999 (09.11.99)  <b>(30) Priority Data:</b> 09/188,607                      9 November 1998 (09.11.98)                      US  <b>(71) Applicant:</b> AGA GAS, INC. [US/US]; 6055 Rockside Woods Boulevard, Cleveland, OH 44131-2329 (US).  <b>(72) Inventor:</b> MACNEAL, James, R.; 16731 Claridon-Troy Road, Burton, OH 44021 (US).  <b>(74) Agents:</b> GARVEY, Michael et al.; Pearne, Gordon, McCoy & Granger LLP, Suite 1200, 526 Superior Avenue, East, Cleveland, OH 44114 (US).		<b>(81) Designated States:</b> AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).  <b>Published</b> <i>With international search report.</i>

**(54) Title:** METHOD FOR REMOVING STERILANT FROM OBJECTS SUBJECTED TO GASEOUS STERILIZATION

**(57) Abstract**

A method of removing sterilization gas from a load (72) disposed in a chamber (24). In accordance with the method, the chamber (24) is evacuated to a subatmospheric pressure. A diffusion gas comprising helium is provided. An amount of the diffusion gas is introduced into the chamber (24) effective to create a superatmospheric diffusion pressure in the chamber (24). The diffusion gas is allowed to diffuse throughout the chamber (24) to displace sterilization gas from the load. The chamber (24) is evacuated to remove portions of the diffusion gas and the sterilization gas from the chamber (24).

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1 METHOD FOR REMOVING STERILANT  
2 FROM OBJECTS SUBJECTED TO GASEOUS STERILIZATION

3 BACKGROUND OF THE INVENTION

4 This invention relates to sterilization in general  
5 and, more particularly, to the removal of sterilant from  
6 objects subjected to gaseous sterilization.

7 Gaseous sterilization is an attractive alternative to  
8 other methods of sterilization, such as steam  
9 sterilization, plasma sterilization, and radiation  
10 sterilization, because gaseous sterilization does not  
11 utilize high temperatures, corrosive chemicals, or high  
12 radiation levels, which can damage objects being  
13 sterilized. Because of these favorable qualities, gaseous  
14 sterilization is commonly used in hospitals to sterilize  
15 medical devices.

16 In gaseous sterilization, objects to be sterilized are  
17 contacted with a gaseous sterilant having good  
18 microbiocidal properties. Ethylene oxide (ETO) is the most  
19 commonly used gaseous sterilant. ETO has excellent  
20 microbiocidal properties, but is extremely volatile and  
21 flammable. The National Fire Protection Association (NFPA)  
22 has given ETO the highest possible flammability hazard  
23 rating under NFPA 704. Since ETO is so volatile and  
24 flammable, an inert gas is often mixed with ETO to suppress  
25 its flammability. Inert gases that are often mixed with  
26 ETO include: carbon dioxide (CO<sub>2</sub>); nitrogen (N<sub>2</sub>);  
27 chlorofluorocarbons (CFCs), such as dichlorodifluoromethane  
28 (CFC-12); hydrochlorofluorocarbons (HCFCs), such as  
29 chlorodifluoromethane (HCFC-22), and monochloro-  
30 tetrafluoroethane, which exists in two isomeric forms, 1-  
31 chloro-1,2,2,2-tetrafluoroethane (HCFC-124), 1-chloro-  
32 1,1,2,2-tetrafluoroethane (HCFC-124a); and mixtures of the  
33 foregoing.

1           For many years, the most commonly used flammability  
2 suppressed ETO mixture was a mixture of 12% ETO and 88%  
3 CFC-12 (commonly referred to as the "12/88 mixture"). Due  
4 to environmental concerns, however, the use of CFCs is  
5 being phased out under the Montreal Protocol. Accordingly,  
6 flammability suppressed ETO mixtures using HCFCs are  
7 becoming more predominant. An example of such a  
8 flammability suppressed ETO mixture using HCFCs is  
9 disclosed in U.S. Patent No. 5,376,333 to Shankland et al.,  
10 which is incorporated herein by reference. Shankland  
11 discloses a suppressed ETO mixture comprising 3 to 13  
12 weight percent ETO and 87 to 97 weight percent of  
13 monochlorotetrafluorethane. Another example of a  
14 flammability suppressed ETO mixture includes a mixture  
15 comprising about 10 weight percent ETO and about 90 weight  
16 percent of a mixture of HCFC-124 and HCFC-22.

17           In a typical gaseous sterilization process utilizing  
18 ETO or an ETO mixture as the sterilant, a load to be  
19 sterilized is first placed in a sterilization chamber. The  
20 chamber is hermetically sealed and a vacuum is drawn to  
21 remove air from the chamber. The chamber is heated and  
22 water vapor is introduced into the chamber, as needed, to  
23 bring the chamber to an optimal relative humidity. The  
24 sterilant is then introduced into the chamber. The load is  
25 exposed to the sterilant for a sterilization period of  
26 time, which is typically between 1 and 6 hours, depending  
27 on the concentration of sterilant and the temperature of  
28 the chamber.

29           After the sterilization period of time, the load is  
30 aerated to remove the sterilant therefrom. Depending on  
31 the construction and capabilities of the sterilizer, the  
32 load is either aerated in the chamber or in a separate  
33 aerator. If the load is composed of a porous material,  
34 such as plastic, or ceramic, the load must be aerated for a  
35 prolonged detoxification or aeration period of time. With  
36 a material such as polyvinylchloride (PVC), the aeration  
37 period of time with current technology is typically between

1 8 and 24 hours, depending on the intended use of the load.  
2 As can be appreciated, such a long period of time is  
3 undesirable because the sterilizer and the load cannot be  
4 re-used during that period of time.

5 Methods have been developed to reduce the aeration  
6 period of time in ETO sterilization processes. An example  
7 of such a method is disclosed in U.S. Patent No. 4,770,851  
8 to Joslyn, which is incorporated herein by reference. In  
9 the Joslyn aeration method, a sterilization chamber  
10 containing a load is evacuated to a subatmospheric pressure  
11 after a sterilization cycle is complete. Steam is then  
12 flushed through the chamber, while the subatmospheric  
13 pressure is maintained in the chamber. The chamber is then  
14 pressurized with heated air, thereby causing some of the  
15 steam to condense on interstices of the load. The chamber  
16 is then evacuated again to the subatmospheric pressure,  
17 thereby causing the steam to vaporize and carry away  
18 residual sterilant from the load.

19 The Joslyn aeration method is a substantial  
20 improvement over conventional aeration methods. Typically,  
21 the Joslyn aeration method reduces the aeration period of  
22 time for PVC to between 4 and 8 hours, depending on the  
23 intended use of the load. This period of time, however, is  
24 still substantial, and certain types of materials may be  
25 damaged by condensing steam on their interstices.

26 Based upon the foregoing, there is a need in the art  
27 for an improved method of removing sterilization gas from a  
28 load. The present invention is directed to such a method.

#### 29 SUMMARY OF THE INVENTION

30 It therefore would be desirable, and is an advantage  
31 of the present invention, to provide an improved method of  
32 removing sterilization gas from a load disposed in a  
33 chamber. In accordance with the method, the chamber is  
34 evacuated to a subatmospheric pressure. A diffusion gas is  
35 provided. An amount of the diffusion gas is introduced  
36 into the sterilization chamber. The amount of the

1 diffusion gas is allowed to diffuse throughout the  
2 sterilization chamber, thereby causing sterilization gas to  
3 diffuse away from the load. Portions of the diffusion gas  
4 and sterilization gas are then removed from the  
5 sterilization chamber.

6 In one embodiment of the present invention, the  
7 diffusion gas is non-flammable and non-condensable at 0-32  
8 psig. In another embodiment, the diffusion gas comprises  
9 helium and the steps of introducing the diffusion gas  
10 through removing the diffusion gas are repeated until an  
11 acceptable residue level of sterilization gas on the load  
12 is attained.

13 Also provided in accordance with the present invention  
14 is a method of sterilizing a load in a sterilization  
15 chamber using sterilization gas. In accordance with the  
16 method, the load is placed in the sterilization chamber and  
17 is exposed to sterilization gas. The sterilization chamber  
18 is evacuated to a subatmospheric pressure, thereby removing  
19 sterilization gas. A diffusion gas is provided that is  
20 selected from the group consisting of helium, hydrogen,  
21 nitrogen, argon, and carbon dioxide and mixtures thereof.  
22 The diffusion gas is introduced into the sterilization  
23 chamber in an amount effective to create a superatmospheric  
24 diffusion pressure in the sterilization chamber. The  
25 amount of the diffusion gas is allowed to diffuse  
26 throughout the sterilization chamber, thereby causing  
27 sterilization gas to diffuse away from the load. Portions  
28 of the diffusion gas and sterilization gas are then removed  
29 from the sterilization chamber.

#### 10 BRIEF DESCRIPTION OF THE DRAWINGS

11 The features, aspects, and advantages of the present  
12 invention will become better understood with regard to the  
13 following description, appended claims, and accompanying  
14 drawings where:

15 Fig. 1 shows a schematic representation of a  
16 sterilization system having an interior chamber; and

1           Fig. 2 shows a graphic representation of a process for  
2 removing sterilant from a load, with time on the y-axis and  
3 pressure of the interior chamber on the x-axis.

#### 4           DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

5           It should be noted that in the detailed description  
6 which follows, parts are parts by weight and percents are  
7 weight percents unless otherwise indicated or apparent.  
8 When a preferred range such as 5-25 is given, this means  
9 preferably at least 5 and preferably not more than 25. It  
10 should also be noted that in order to clearly and concisely  
11 disclose the present invention, the drawings may not  
12 necessarily be to scale and certain features of the  
13 invention may be shown in somewhat schematic form.

14           Referring now to Fig. 1, there is shown a  
15 sterilization system 10 wherein the present invention may  
16 be practiced. The sterilization system 10 may be a  
17 commercially available ETO sterilization system that has  
18 been modified to practice the present invention. The  
19 sterilization system 10 generally includes a sterilization  
20 vessel 12, a sterilant source 14, a sterilant supply system  
21 16, a sterilant removal system 18, and a gas diffusion  
22 system 20. The sterilization system 10 may also optionally  
23 include a steam supply system 22.

24           The sterilization vessel 12 is preferably composed of  
25 stainless steel and defines an interior chamber 24 having  
26 an open end. A door 26 is pivotably mounted to the  
27 sterilization vessel 12 and is pivotable between an open  
28 position, wherein the door 26 is spaced from the open end,  
29 and a closed position, wherein the door 26 covers the open  
30 end 24. A conventional lock assembly (not shown) is  
31 provided to lock the door 26 in the closed position. The  
32 door 26 and the sterilization vessel 12 are provided with  
33 seals 27, which cooperate to hermetically seal the open  
34 end when the door 26 is locked in the closed position. A  
35 circulation fan 28 may be mounted to the sterilization  
36 vessel 12 to provide circulation and uniform environmental

1 conditions in the interior chamber 24. A pressure  
2 transducer 29 may be connected to the sterilization vessel  
3 by a conduit 30.

4 A heating system is provided to heat the interior  
5 chamber 24. The heating system includes a heating device  
6 31 disposed around the exterior of the sterilization vessel  
7 12, and a control device (not shown). The heating device  
8 31 may be an electric resistance heating coil, or other  
9 type of heating means, such as a hot water or steam jacket.  
10 The control device regulates the flow of electricity or hot  
11 water or steam through the heating device 31, or otherwise  
12 controls the heating device 31, so as to maintain the  
13 interior chamber 24 at a selected temperature.

14 The sterilant source 14 preferably includes a pair of  
15 pressurized tanks 32 for holding a sterilant under  
16 pressure. The sterilant may be 100% ETO, the 12/88  
17 mixture, or a mixture of about 8-12% ETO and about 88-92%  
18 CO<sub>2</sub>, or N<sub>2</sub>. More preferably, the sterilant is a mixture of  
19 about 3-13% ETO and about 87-97% HCFC. Still more  
20 preferably, the sterilant is a mixture of about 9-12% ETO  
21 and about 88-91% nonochlorotetrafluoroethane (the  
22 "ETO/HCFC-124 mixture"). Still more preferably, the  
23 sterilant is a mixture of about 10 weight percent ETO and  
24 about 90 weight percent of a mixture of HCFC-124 and HCFC-  
25 22 (the "ETO/HCFC-124/HCFC-22 mixture"), which is available  
26 from Allied Signal under the name Oxyfume 2002, and from  
27 the Pennsylvania Engineering Company under the name Penngas  
28 2.

29 The tanks 32 are preferably disposed in a sealed  
30 enclosure 34 connected to a ventilation system (not shown)  
31 that maintains the enclosure 34 at a slightly negative  
32 pressure. The tanks 32 are pressurized to maintain the  
33 sterilant in liquid form. If the sterilant is the  
34 ETO/HCFC-124/HCFC-22 mixture, the tanks 32 are pressurized  
35 to about 60 psig. Eductor tubes (not shown) are disposed  
36 in the tanks 32 to conduct the sterilant from the bottoms  
37 of the tanks 32 to a header assembly 36, which connects the



1 tanks 32 to the sterilant supply system 16. The sterilant  
2 is supplied from the tanks 32 sequentially such that the  
3 sterilant is supplied from only one of the tanks 32 at a  
4 time. Preferably, the header assembly 36 is provided with  
5 an automatic transfer feature that automatically switches  
6 from an exhausted one of the tanks 32 to a filled one of  
7 the tanks 32, without interrupting the supply of sterilant.

8 The sterilant supply system 16 supplies the sterilant  
9 from the sterilant source 14 to the sterilization vessel  
10 12. The sterilant supply system 16 includes a vaporizer 38  
11 having an inlet connected to the header assembly 36 by  
12 piping 40, and an outlet connected to the sterilization  
13 chamber by piping 41. A solenoid valve 42 is disposed in  
14 the piping 40 and is operable to control the supply of  
15 sterilant to the vaporizer 38 and, thus, the sterilization  
16 vessel 12. The vaporizer 38 reduces the pressure of the  
17 sterilant and heats the sterilant, thereby causing the  
18 sterilant to vaporize into a gas. The vaporizer 38 is  
19 controlled such that the temperature of the sterilant gas  
20 entering the sterilization vessel 12 is at a predetermined  
21 temperature that will not exceed the temperature limit of  
22 the load being sterilized.

23 The steam supply system 22 may be provided to supply  
24 steam to the sterilization vessel 12 prior to sterilization  
25 in order to raise the humidity in the interior chamber 24  
26 and hydrate microorganisms on the load disposed therein.  
27 If the sterilization system 10 is based on an ETO  
28 sterilization system obtained from or modified by the  
29 Joslyn Sterilizer Corporation, the steam supply system 22  
30 may also be used to remove air from the interior chamber 24  
31 pursuant to an air removal method disclosed in U.S. Patent  
32 No. 4,770,851 to Joslyn, referenced earlier. The air  
33 removal method of Joslyn is similar to the Joslyn aeration  
34 method described above and utilizes a plurality of  
35 alternating steam and pressurized air pulses.

36 In lieu of using steam to raise the humidity in the  
37 interior chamber 24, other conventional humidifying means

1        may be employed. For example, a moisture-releasing device  
2        may be placed in the interior chamber 24 along with the  
3        load to be sterilized. An example of such a moisture-  
4        releasing device is disclosed in U.S. Patent No. 5,135,715  
5        to Andersen, which is incorporated herein by reference.

6        Gaseous sterilization with ETO is more effective in  
7        killing microorganisms if the microorganisms are hydrated  
8        and if the sterilization process is carried out in an  
9        atmosphere having at least 30% relative humidity. Thus,  
10       the humidifying means chosen should be able to maintain the  
11       interior chamber 24 at a relative humidity of at least 30%.

12       The gas removal system 18 removes gas from the  
13       interior chamber 24. The gas removal system 18 includes a  
14       vacuum pump 44 having an inlet connected to the  
15       sterilization vessel 12 by piping 46, and an outlet  
16       connected to a vent 48 by piping 50. A solenoid valve 52  
17       is disposed in the piping 46 and is operable to control the  
18       removal of gas from the interior chamber 24. The vacuum  
19       pump 44 may be an open water sealed vacuum pump, or more  
20       preferably, a dry vacuum pump, or a recycled sealing fluid  
21       vacuum pump. Preferably, the operation of the vacuum pump  
22       44 and the solenoid valve 52 is controlled by a  
23       programmable controller 54.

24       In accordance with the present invention, the gas  
25       diffusion system 20 cooperates with the gas removal system  
26       18 to remove gaseous sterilant from the interior chamber 24  
27       after sterilization. The gas diffusion system 20 includes  
28       at least one tank 56, or more preferably, a pair of tanks  
29       56 of a compressed diffusion gas.

30       As will become more apparent below, it is desirable  
31       for the diffusion gas to have a fast rate of diffusion.  
32       The rate of diffusion of a gas is inversely proportional to  
33       the square root of its molecular weight. In addition, the  
34       rate of diffusion of a gas is proportional to temperature  
35       and the negative gradient of the density of the gas. Thus,  
36       it is desirable for the diffusion gas to be light, i.e.,  
37       have as low a molecular weight as possible, and to

1     introduce the diffusion gas at an increased temperature and  
2     pressure. It is also preferable if the diffusion gas is  
3     not an oxidizer, is unable to support combustion, and is  
4     non-flammable. It is further preferable if the diffusion  
5     gas is non-condensable at 0-32 psig, more preferably 0-50  
6     psig, and is inert. Since helium is the lightest non-  
7     flammable gas, is non-condensable at 0-50 psig, and is  
8     inert, the diffusion gas is preferably about 100 percent  
9     helium, more preferably 100% United States Pharmacopeia  
10    (USP) helium, i.e., medical grade helium. Less preferably,  
11    the diffusion gas is a mixture of about 80-90 percent  
12    helium and about 10-20 percent hydrogen (H<sub>2</sub>). Less  
13    preferably, the diffusion gas is a mixture of about 51-99  
14    percent helium and about 1-49 percent of a gas selected  
15    from the group consisting of hydrogen, nitrogen (N<sub>2</sub>), argon  
16    (Ar), carbon dioxide (CO<sub>2</sub>), air, and mixtures thereof,  
17    wherein the amount of hydrogen present is insufficient to  
18    make the diffusion gas flammable, which is about 20 percent  
19    or less. Less preferably, the diffusion gas is a mixture  
20    of about 1-50 percent helium and about 50-99 percent of a  
21    gas selected from the group consisting of hydrogen,  
22    nitrogen, argon, carbon dioxide, air, and mixtures thereof,  
23    wherein the amount of hydrogen present is insufficient to  
24    make the diffusion gas flammable. Less preferably, the  
25    diffusion gas is about 50-100 percent nitrogen and about 0-  
26    50 percent of a gas selected from the group consisting of  
27    hydrogen, argon, carbon dioxide, air, and mixtures thereof,  
28    wherein the amount of hydrogen present is insufficient to  
29    make the diffusion gas flammable. Less preferably the  
30    diffusion gas is a mixture of about 50-100 percent air and  
31    about 0-50 percent of a gas selected from the group  
32    consisting of hydrogen, argon, carbon dioxide, and mixtures  
33    thereof, wherein the amount of hydrogen present is  
34    insufficient to make the diffusion gas flammable. Less  
35    preferably, the diffusion gas is a mixture of about 50-100  
36    percent CO<sub>2</sub> and about 0-50 percent of a gas selected from  
37    the group consisting of hydrogen, argon, air, and mixtures

1       thereof, wherein the amount of hydrogen present is  
2       insufficient to make the diffusion gas flammable.

3             If the diffusion gas is helium, the tanks 56 are  
4       pressurized to about 2,200 psig. The tanks 56 have  
5       conventional outlet assemblies that include shutoff valves  
6       58. The outlet assemblies of the tanks 56 are connected to  
7       a header assembly 60 having pressure reducing devices 62.  
8       The diffusion gas is supplied from the tanks 56  
9       sequentially such that the diffusion gas is supplied from  
10      only one of the tanks 56 at a time. Preferably, the header  
11      assembly 60 has an automatic transfer feature that  
12      automatically switches from an exhausted one of the tanks  
13      56 to a filled one of the tanks 56, without interrupting  
14      the supply of diffusion gas.

15            The header assembly 60 may be connected by piping 66  
16      to a bioretentive filter 63 that removes any bacteria,  
17      viruses, or fungi that may be present in the diffusion gas.  
18      An inlet of a heater 64 is connected to the filter 63 by  
19      piping 65. An outlet of the heater 64 is connected to the  
20      sterilization vessel 12 by piping 68. A solenoid valve 70  
21      is disposed in the piping 66 and is operable to control the  
22      flow of the diffusion gas to the filter 63 and, thus, the  
23      heater 64 and the interior chamber 24. The operation of  
24      the solenoid valve 70 is preferably controlled by the  
25      programmable controller 54.

26            In order to sterilize a load 72 using the  
27      sterilization system 10, the load 72 is preferably wrapped,  
28      packaged, or otherwise covered with a protective material  
29      74 to form a protected space 76 within which the load 72 is  
30      disposed. The protective material 74 may be muslin, paper,  
31      plastic or other material specially designed to maintain or  
32      preserve the sterility of the load 72 after removal from  
33      the sterilization system 10. The load 72 may be pre-  
34      packaged with the protective material 74 by the  
35      manufacturer of the load 72, or the load 72 may be wrapped  
36      or packaged with the protective material 74 at the site  
37      where the sterilization system 10 is located.

1           The door 26 is moved to the open position and the  
2           packaged load 72 is placed in the interior chamber 24. The  
3           door 26 is then closed and locked. The heating device 31  
4           is activated to heat the interior chamber 24 to a  
5           sterilization temperature in a range from about 20°C to  
6           about 100°C, depending upon the nature of the load 72.  
7           Preferably, the sterilization temperature is in a range  
8           from about 30°C to about 60°C, more preferably, about 54°C  
9           (130°F). The relative humidity of the interior chamber 24  
10          is raised above 30 percent, more preferably above 65  
11          percent. Air is removed from the interior chamber 24  
12          before, during or after the humidification. The air is  
13          removed by opening the solenoid valve 52 and actuating the  
14          vacuum pump 44 until the interior chamber 24 has a vacuum  
15          or negative gauge pressure P1 of about 20-25 inches of  
16          mercury. If the sterilization system 10 is based on an ETO  
17          sterilization system obtained from the Joslyn Sterilizer  
18          Corporation, the alternating steam and pressurized air  
19          pulses of the Joslyn air removal method may also be  
20          employed to remove the air and humidify the interior  
21          chamber 24.

22          The solenoid valve 42 is actuated to cause the  
23          sterilant to flow through the piping 40 to the vaporizer  
24          38, where the sterilant is vaporized and heated to the  
25          sterilization temperature, which, as set forth above, is  
26          preferably about 54°C. The gaseous sterilant flows from  
27          the vaporizer 38 into the interior chamber 24 through the  
28          piping 41. The gaseous sterilant is admitted into the  
29          interior chamber 24 in an amount that preferably produces a  
30          pressure P2 in the interior chamber 24, which is greater  
31          than atmospheric pressure, i.e., is superatmospheric.  
32          Preferably, the pressure P2 is in a range from about .1  
33          psig to about 32 psig, more preferably about 12 psig. The  
34          load 72 is maintained in the interior chamber 24 for a  
35          sterilization period of time, which may be between 1 and 6  
36          hours.

1           At the conclusion of the sterilization period of time,  
2           the sterilant removal method of the present invention is  
3           performed to remove the sterilant from the interior chamber  
4           24 and the load 72. Preferably, the sterilant removal  
5           method is controlled by the programmable controller 54.

6           Referring now to Fig. 2, the interior chamber 24 is  
7           evacuated to the pressure P1 by opening the solenoid valve  
8           52 and actuating the vacuum pump 44. This evacuation is  
9           maintained between 80 and 82 and removes most of the  
10          gaseous sterilant from the interior chamber 24. At 82, the  
11          solenoid valve 52 is closed and the vacuum pump 44 is  
12          deactivated.

13          Referring back to Fig. 1, the solenoid valve 70 is  
14          actuated to cause the diffusion gas to flow through the  
15          piping 66 to the filter 63 and thence the heater 64, which  
16          is activated to heat the diffusion gas to a diffusion  
17          temperature T2. Preferably, T2 is above room temperature  
18          so as to speed up the diffusion rate of the diffusion gas.  
19          Preferably T2 is in a range from about 30°C to about 70°C,  
20          more preferably, about 60°C (140°F). The diffusion gas  
21          flows from the heater 64 into the interior chamber 24  
22          through the piping 68. The diffusion gas is admitted into  
23          the interior chamber 24 until the pressure in the interior  
24          chamber 24 reaches a diffusion pressure P3 as shown at 84  
25          of Fig. 2. Preferably, P3 is superatmospheric so as to  
26          increase the diffusion rate of the diffusion gas, and is  
27          greater than the sterilization pressure P2. Preferably, P3  
28          is in a range from about .1 psig to about 50 psig, more  
29          preferably in a range from about 5 psig to about 32 psig,  
30          more preferably in a range from greater than 12 psig to  
31          about 25 psig. Preferably, the heating device 31 is  
32          manipulated to maintain the temperature of the interior  
33          chamber 24 at the diffusion temperature T2.

34          The diffusion gas is allowed to diffuse throughout the  
35          interior chamber 24. Since the diffusion gas is preferably  
36          a light gas, the diffusion gas readily passes through the  
37          protective material 74, even if it is composed of plastic.

1 The diffusion gas enters the protected space 76 within  
2 which the load 72 is located or disposed and moves into the  
3 interstices of the load 72. If permitted, the diffusion  
4 gas will continue to diffuse into the protected space 76  
5 and the interstices of the load 72 until the concentration  
6 of the diffusion gas is uniform throughout the protected  
7 space 76, the interstices of the load 72, and the rest of  
8 the interior chamber 24.

9 In order to maintain a uniform gas concentration  
10 throughout the interior chamber 24, the diffusion of the  
11 diffusion gas into the protected space 76 and the  
12 interstices of the load 72 is accompanied by the diffusion  
13 of sterilant out of the interstices of the load 72 and the  
14 protected space 76 and into the remaining portion of the  
15 interior chamber 24. If permitted, this opposing diffusion  
16 of sterilant will continue until the concentration of  
17 sterilant is uniform throughout the interior chamber 24, at  
18 which point a substantial portion of the sterilant will  
19 have been removed from the protected space 76 and the  
20 interstices of the load 72.

21 Referring back to Fig. 2, the foregoing diffusion  
22 period is maintained between 84 and 86 to allow the  
23 diffusion gas to diffuse into the protected space 76 and  
24 the interstices and thereby displace sterilant. At the  
25 conclusion of the diffusion period, the solenoid valve 52  
26 is opened and the vacuum pump 44 is actuated to evacuate  
27 the interior chamber 24 to the pressure P1 for a period  
28 between 88 and 90. The evacuation of the interior chamber  
29 24 removes most of the helium and remaining sterilant from  
30 the interior chamber 24.

31 The process from 82 to 90 is repeated if and until an  
32 acceptable sterilant residue level is attained as shown at  
33 92. The duration and number of repetitions of the process  
34 may vary based on the use of the load 72 because the  
35 acceptable sterilant residue level is dependent upon the  
36 use of the load 72. If the load 72 is for contact with  
37 skin or mucosa, the acceptable sterilant residue level is

1 about 250 ppm, whereas if the load 72 is for implantation  
2 or contact with blood or tissue, the acceptable sterilant  
3 residue level is about 25 ppm.

4 The duration and number of repetitions of the process  
5 from 82 to 90 may also vary based on the composition of the  
6 load 72 and the protective material 74. The rate of  
7 diffusion of a gas is much slower through materials such as  
8 plastic (and in particular, PVC) than it is through  
9 materials, such as cloth and paper.

10 When the acceptable sterilant residue level is  
11 attained, the pressure of the interior chamber 24 is vented  
12 to atmospheric pressure as shown at 94. The load 72 is  
13 then removed from the interior chamber 24 and used as  
14 needed.

15 While the invention has been shown and described with  
16 respect to a particular embodiment thereof, this embodiment  
17 is for the purpose of illustration rather than limitation,  
18 and other variations and modifications of the specific  
19 embodiment herein described will be apparent to those  
20 skilled in the art, all within the intended spirit and  
21 scope of the invention. For example, the removal of the  
22 residual sterilant from the load 72 using the diffusion gas  
23 may be performed in a separate aerator rather than in the  
24 sterilization vessel 12.

25 Accordingly, the invention is not to be limited in  
26 scope and effect to the specific embodiment herein  
27 described, nor in any other way that is inconsistent with  
28 the extent to which the progress in the art has been  
29 advanced by the invention.



**WHAT IS CLAIMED IS:**

1           1. A method of removing sterilization gas from a load  
2 disposed in a chamber, said method comprising the steps of:  
3           evacuating the chamber to a subatmospheric pressure;  
4           providing a diffusion gas that is non-flammable and  
5 non-condensable at 0-32 psig;  
6           introducing an amount of the diffusion gas into the  
7 chamber;  
8           allowing the amount of the diffusion gas to diffuse  
9 throughout the chamber, thereby causing sterilization gas  
10 to diffuse away from the load; and  
11          removing portions of the diffusion gas and  
12 sterilization gas from the chamber.

1           2. The method of claim 1, wherein the diffusion gas  
2 is about 100 weight percent helium.

1           3. The method of claim 1, wherein the diffusion gas  
2 comprises about 80-85 weight percent helium and about 15-20  
3 weight percent hydrogen.

1           4. The method of claim 1, wherein the diffusion gas  
2 comprises about 51-99 weight percent helium and about 1-49  
3 weight percent of a gas selected from the group consisting  
4 of hydrogen, nitrogen, argon, carbon dioxide, air, and  
5 mixtures thereof.

1           5. The method of claim 1, wherein the amount of the  
2 diffusion gas introduced into the chamber is effective to  
3 create a superatmospheric diffusion pressure in the  
4 chamber.

1           6. The method of claim 5, wherein the diffusion  
2 pressure is about 5-32 psig.

1           7. The method of claim 6, further comprising the step  
2 of heating the diffusion gas to a diffusion temperature of  
3 about 30-70°C.

1           8. The method of claim 1, wherein the step of  
2 removing portions of the diffusion gas and sterilization  
3 gas from the chamber comprises evacuating the chamber to  
4 the subatmospheric pressure.

1           9. The method of claim 8, further comprising the  
2 steps of:  
3           flushing steam through the chamber, while maintaining  
4 the subatmospheric pressure in the chamber;  
5           pressurizing the chamber with heated air; and  
6           removing heated air from the chamber by evacuating the  
7 chamber to the subatmospheric pressure.

1           10. The method of claim 9, wherein the steps of  
2 flushing with steam, pressurizing with heated air, and  
3 removing heated air are performed before the step of  
4 introducing the diffusion gas into the chamber.

1           11. The method of claim 9, wherein the steps of  
2 flushing with steam, pressurizing with heated air, and  
3 removing heated air are performed after the step of  
4 removing portions of the diffusion gas from the chamber.

1           12. The method of claim 8, further comprising the  
2 steps of:  
3           introducing a second amount of the diffusion gas into  
4 the chamber after the step of removing portions of the  
5 diffusion gas and sterilization gas from the chamber; and  
6           allowing the second amount of the diffusion gas to  
7 diffuse throughout the chamber, thereby causing  
8 sterilization gas to diffuse away from the load.

1           13. The method of claim 8, wherein the subatmospheric  
2           pressure is about 20-25 inches of mercury.

1           14. The method of claim 1, wherein the sterilization  
2           gas comprises ethylene oxide.

1           15. A method of sterilizing a load in a sterilization  
2           chamber using sterilization gas, said method comprising the  
3           steps of:

4           placing the load in the sterilization chamber;  
5           exposing the load to sterilization gas;  
6           evacuating the sterilization chamber to a  
7           subatmospheric pressure, thereby removing sterilization  
8           gas;

9           providing a diffusion gas selected from the group  
10          consisting of helium, hydrogen, nitrogen, argon, and carbon  
11          dioxide and mixtures thereof;

12          introducing the diffusion gas into the sterilization  
13          chamber in an amount effective to create a superatmospheric  
14          diffusion pressure in the sterilization chamber;

15          allowing the amount of the diffusion gas to diffuse  
16          throughout the sterilization chamber, thereby causing  
17          sterilization gas to diffuse away from the load; and

18          removing portions of the diffusion gas and  
19          sterilization gas from the sterilization chamber.

1           16. The method of claim 15, wherein the diffusion  
2           pressure is in a range from about .1 psig to about 50 psig.

1           17. The method of claim 16, wherein the diffusion  
2           pressure is about 5-32 psig.

1           18. The method of claim 17, further comprising the  
2           step of heating the diffusion gas to a diffusion  
3           temperature of about 30-70°C.

1           19. The method of claim 15, wherein the diffusion gas  
2           is about 100 weight percent helium.

1           20. A method of removing sterilization gas from a  
2           load disposed in a chamber, said method comprising the  
3           steps of:  
4           (a) evacuating the chamber to a subatmospheric  
5           pressure;  
6           (b) providing a diffusion gas comprising helium;  
7           (c) introducing an amount of the diffusion gas into  
8           the chamber;  
9           (d) allowing the amount of the diffusion gas to  
10          diffuse throughout the chamber, thereby causing  
11          sterilization gas to diffuse away from the load;  
12          (e) removing portions of the diffusion gas and  
13          sterilization gas from the chamber; and  
14          (f) repeating steps (c) through (e) until an  
15          acceptable residue level of sterilization gas on the load  
16          is attained.

1           21. The method of claim 20, wherein the diffusion gas  
2           is about 100 weight percent helium.

1           22. The method of claim 21, wherein the amount of the  
2           diffusion gas introduced into the chamber is effective to  
3           create a diffusion pressure of about 5-32 psig in the  
4           chamber.

1           23. The method of claim 22, further comprising the  
2           step of heating the diffusion gas to a diffusion  
3           temperature of about 30-70°C.

1           24. The method of claim 23, wherein the sterilization  
2           gas comprises a mixture of about 9-12% ethylene oxide and  
3           about 88-91% monochlorotetrafluoroethane.

1           25. The method of claim 23, wherein the sterilization  
2 gas comprises a mixture of about 9-12% ethylene oxide and a  
3 mixture of about 88-91% monochlorotetrafluoroethane and  
4 chlorodifluoromethane.

1           26. The method of claim 20, wherein the diffusion gas  
2 comprises about 51-99 weight percent helium and about 1-49  
3 weight percent of a gas selected from the group consisting  
4 of hydrogen, nitrogen, argon, carbon dioxide, air, and  
5 mixtures thereof.

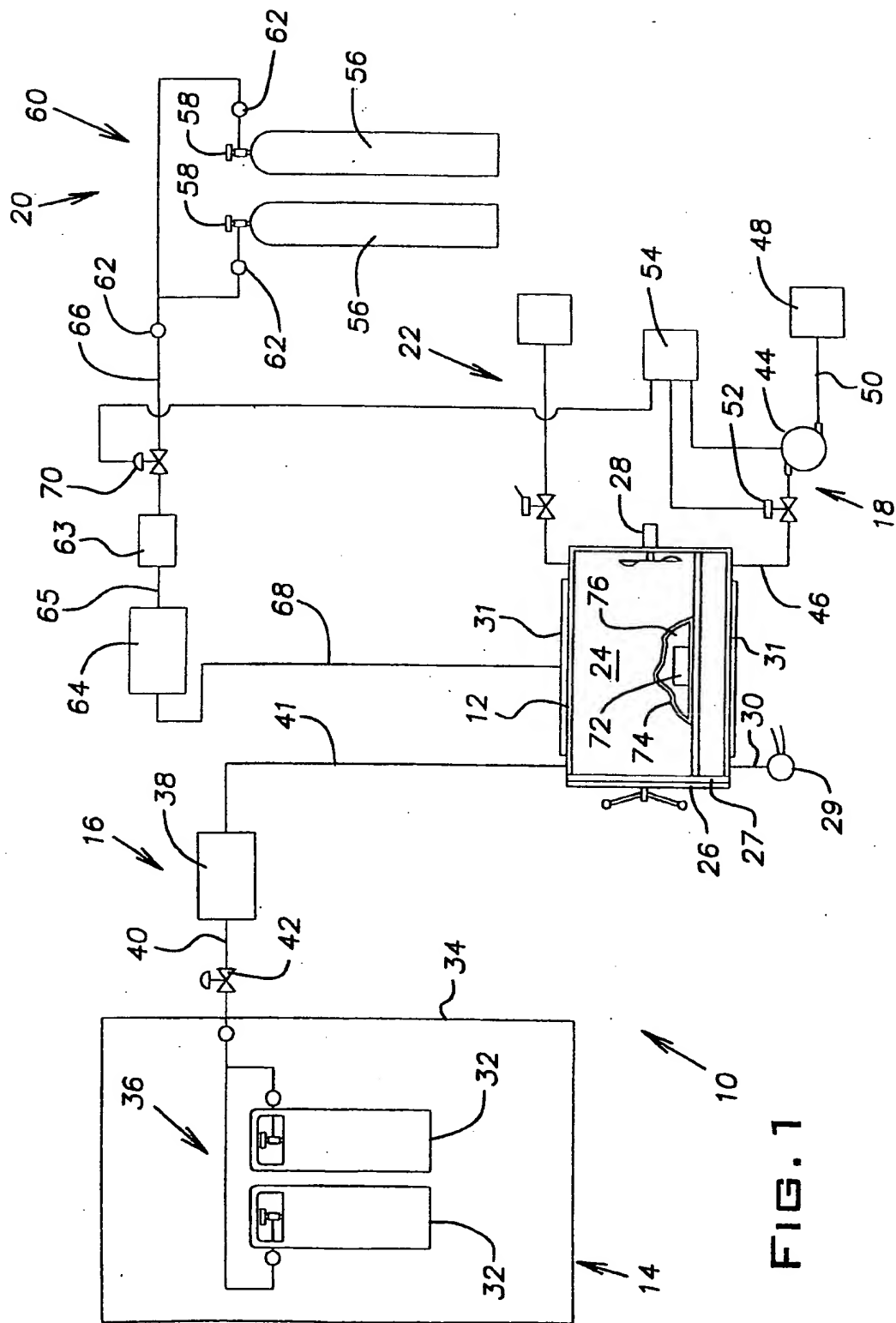


FIG. 1

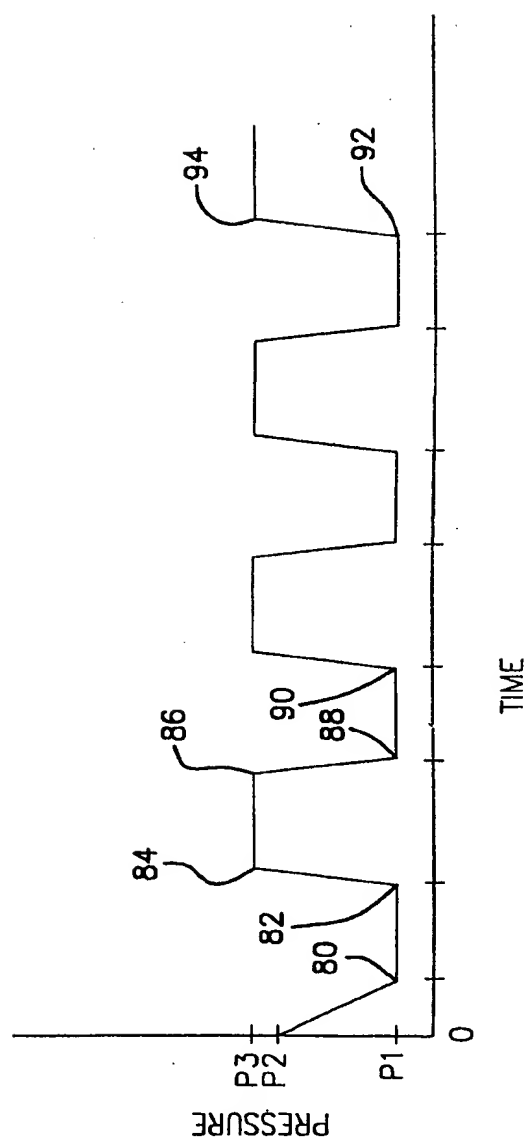


FIG. 2

## INTERNATIONAL SEARCH REPORT

International application No.

PCT/US99/26367

## A. CLASSIFICATION OF SUBJECT MATTER

IPC(6) : A61L 2/20

US CL : 422/28, 1, 26, 27, 33, 34

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 422/28, 1, 26, 27, 33, 34

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EAST, search terms: ethylene oxide, decontamination, air, helium

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 5,128,101 A (BOYNTON) 07 July 1992, whole document, especially col. 1.	1-26
Y	US 5,376,333 A (SHANKLAND et al) 27 December 1994, whole document, especially col. 4.	1-26
Y	US 5,830,409 A (CHILDERS et al) 03 November 1998, whole document, especially columns 1-2.	1-26
A	US 4,130,393 A (FOX) 19 December 1978, whole document.	1-26



Further documents are listed in the continuation of Box C.



See patent family annex.

Special categories of cited documents:		"T"	later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"A"	document defining the general state of the art which is not considered to be of particular relevance		
"B"	earlier document published on or after the international filing date	"X"	document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
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"O"	document referring to an oral disclosure, use, exhibition or other means		
"P"	document published prior to the international filing date but later than the priority date claimed	"A"	document member of the same patent family

Date of the actual completion of the international search

09 FEBRUARY 2000

Date of mailing of the international search report

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